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ELI LILLY AND COMPANY

By Linda M. Dunham

Date May 7, 2004

**PATENT APPLICATION**  
**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**  
**Before the Board of Patent Appeals and Interferences**

Appellants:	William Webster Thompson et al	) Confirmation No.:
		) 2652
Serial No.:	10/048,239	)
		) Group Art Unit:
Filed:	04/30/2002	) 1623
		)
For:	ECTOPARASITICIDAL AQUEOUS	)
	SUSPENSION FORMULATIONS OF	)
	SPINOSYNS	) Examiner:
Docket No.:	X-12636	) Leigh C. Maier

**BRIEF FOR THOMPSON ET AL.**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450  
Sir:

Appellants appeal from the final rejection dated December 11, 2003, of claims 2-4, 8-10, 12, 13 and 16 of this application.

**Real Party in Interest**

The real party in interest is Eli Lilly and Company, the assignee of the entire right, title and interest in this application.

**Related Appeals and Interferences**

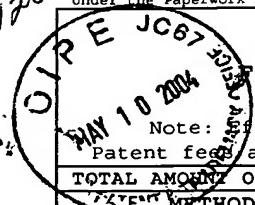
There are no related appeals or interferences.

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IFW AF/1623  
PTO/SB/17 (12/97)

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Note: Effective November 10, 1998.  
Patent fees are subject to annual revision.

**TOTAL AMOUNT OF PAYMENT** (\$ 330.00)

**METHOD OF PAYMENT** (check one)

1.  The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:

Deposit Account Number 05-0840

Deposit Account Name Eli Lilly and Company

- Charge Any Additional Fee Required Under 37 CFR 1.16 and 1.17  Charge the Issue Fee Set in 37 CFR 1.18 at the Mailing of the Notice of Allowance

2.  Payment Enclosed:

Check  Money Order  Other

## FEE CALCULATION

### 1. FILING FEE

Large Fee Code	Entity Fee (\$)	Small Fee Code	Entity Fee (\$)	Fee Description	Fee Paid
101	770	201	385	Utility filing fee	
106	340	206	170	Design filing fee	
107	530	207	265	Plant filing fee	
108	770	208	385	Reissue filing fee	
114	160	214	80	Provisional filing fee	
<b>SUBTOTAL (1)</b>		(\$ 0)			

### 2. CLAIMS

Total Claims	-20**=	Extr a	Fee from below	Fee Paid
Independent Claims	-3**=		X 18	=
Multiple Dependent Claims (first time)			X 86	=
			290	=

Large Fee Code	Entity Fee (\$)	Small Fee Code	Entity Fee (\$)	Fee Description
103	18	203	9	Claims in excess of 20
102	86	202	43	Independent claims in excess of 3
104	290	204	145	Multiple dependent claim
109	86	209	43	Reissue independent claims over original patent
110	18	210	9	Reissue claims in excess of 20 and over original patent
<b>SUBTOTAL (2)</b>		(\$ 0)		

\*\*or number previously paid, if greater; For Reissues, see above

Complete if Known			
<b>Application Number</b>			10/048,239
<b>Filing Date</b>			April 30, 2002
<b>First Named Inventor</b>			William Webster Thompson
<b>Group Art Unit</b>			1623
<b>Examiner Name</b>			Leigh C. Maier
<b>Attorney Docket Number</b>			X-12636

## FEE CALCULATION (continued)

### 3. ADDITIONAL FEES

Large Fee Code	Entity Fee (\$)	Small Fee Code	Entity Fee (\$)	Fee Description	Fee Paid
105	130	205	65	Surcharge-late filing fee or oath	
127	50	227	25	Surcharge-late provisional filing fee or cover sheet	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	420	216	210	Extension for reply within second month	
117	950	217	475	Extension for reply within third month	
118	1,480	218	740	Extension for reply within fourth month	
128	2,010	228	1,005	Extension for reply within fifth month	
119	330	219	165	Notice of Appeal	
120	330	220	165	Filing a brief in support of an appeal	330
121	290	221	145	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive-unavoidable	
141	1,330	241	665	Petition to revive-unintentional	
142	1,330	242	665	Utility issue fee (or reissue)	
143	480	243	240	Design Issue Fee	
144	640	244	320	Plant Issue Fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Petitions related to provisional applications	
126	180	126	180	Submission of Information Disclosure Stmt.	
581	40	581	40	Recording each patent assignment per property (times number of properties)	
146	770	246	385	Filing a submission after final rejection (37 CFR 1.129(a))	
149	770	249	385	For each additional invention to be examined (37 CFR 1.129(b))	
179	770	279	385	Request for Continued Examination (RCE)	
169	900	169	900	Request for expedited examination of a design application	
Other fee (specify)		Terminal Disclaimer 1.321			
Other fee (specify)					
*Reduced by Basic Filing Fee Paid				<b>SUBTOTAL (3)</b>	<b>(\$ 330.00)</b>

SUBMITTED BY		Complete (if applicable)
Typed Name	John G. Demeter	Reg. Number 30,167
Signature		Date May 7, 2004

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ELI LILLY AND COMPANY

Linda M. Durbin  
By

May 7, 2004  
Date

Status of Claims

Claims 1, 5-7, 11, 14 and 15 have been cancelled.

Claims 2-4, 8-10, 12, 13 and 16 are pending in this Appeal.

Status of Amendments

A proposed amendment filed after the Final Rejection has been acted upon by the Examiner and entered for purposes of this appeal. Following entry of the proposed amendment after Final Rejection, all pending claims (claims 2-4, 8-10, 12, 13 and 16) stand as rejected.

Summary of Invention

The invention defined in the claims on appeal is directed toward a stable ectoparasiticidal aqueous suspension formulation suitable for administration to animals comprising spinosad as active ingredient milled to a specified average particle size; a specified dispersant where the spinosad to dispersant weight ratio is stated; a mineral thickener in a specified weight percent range; an antimicrobial agent; propylene glycol; xanthan gum in a specified weight percent range; a surfactant in a specified weight percent range; an antifoam agent and water. A general description of the formulation can be found at least at page 2, line 18 through page 3, line 24 of the specification.

Claim 10 is directed toward an article of manufacture that includes the formulation described above and claims 12 and 13 are directed toward methods of using the formulation described above. The article of manufacture and method of use claims are generally described at least on page 11, line 24 through page 12, line 27 of the specification.

Issues

Whether claims 2-4, 8-10, 12, 13 and 16 are patentable under 35 U.S.C. § 103(a) over U.S. Patent 5,571,901 (Boeck, et al.) in view of Burton, et al., Proc. – Beltwide Cotton Conf., 2: 696-697 (1996).

Grouping of Claims

All of the claims on appeal stand or fall together.

Case for Appellant(s)

Appellants contend all pending claims are patentable over Boeck et al. in view of Burton et al. The burden is on the Examiner to establish a prima facie case of obviousness. In re Bell, 26 U.S.P.Q. 2d 1529 (Fed. Cir. 1993). A combination of Boeck et al. in view of Burton et al. does not establish a prima facie case of obviousness against the

presently claimed invention because a condensed formaldehyde/naphthalene sulfonic acid or salt thereof dispersant at a spinosad:dispersant weight ratio of 3:1 to 1:5 in an aqueous suspension formulation is not taught or suggested.. Assuming, however, a prima facie case of obviousness is found to exist, Example 1 of the present specification demonstrates the unobviousness and therefore patentability of the presently claimed invention.

The Art

Appellants contend Boeck et al. does not establish a prima facie case of obviousness against the presently claimed invention. A reference which gives only general guidance and is not specific as to the particular form of the claimed invention and how to achieve it does not render the claimed invention unpatentably obvious. In re Baird, 29 U.S.P.Q. 2d 1550 (Fed. Cir. 1994); Ex parte Obukowicz, 27 U.S.P.Q. 2d 1063 (B.P.A.I. 1992). The disclosure in Boeck et al. at column 25, line 3 to column 26, line 40 describes various agricultural compositions that may be prepared with a single A83543 compound, mixture of compounds, the separated A83543 mixture or the crude dried fermentation broth. At column 25, lines 21-27, wettable powder type formulations are described as an intimate mixture of the active compound, an inert carrier and surfactants. The inert carrier is said to usually be chosen from certain clays, diatomaceous earths or purified silicates. At column 25, lines 28-33, surfactants that may be used to provide a wettable powder are described. Included in the description of surfactants are “condensed naphthalenesulfonates” and “naphthalenesulphonates.”

Boeck et al. also includes at column 41, lines 4-9, Example 13C, an exemplary composition termed a suspension consisting of A83543A, a naphthalenesulfonate salt, a nonionic surfactant, fumed silica and water. The weight ratio of spinosyn A to naphthalenesulfonate salt is 6:1 (30%:5%). No further description of the nature or function of the naphthalenesulfonate salt is provided.

Burton et al., discloses a spinosad (spinosyn A and D) containing suspension concentrate formulation where the spinosad has a particle size of between 2 and 6 microns and containing other “inerts” to “balance viscosity, settling characteristics of the concentrate, and dispersion of the diluted product.” No further details are provided on the type, nature or function of the inert materials in the formulation. Burton et al., discloses data on the formulation of density over temperature ranges and time; viscosity over freeze/thaw and heated cycling; stability over time and temperature; and suspension/resuspension characteristics.

Deficiency of the Art

As stated on page 2, lines 3-10 of the present specification, the inclusion of a dispersant and the ratio of active ingredient to dispersant is a unique feature of the

formulations of the presently claimed invention.

Appellants contend neither Boeck et al., nor Burton et al., alone or in combination, suggests the requirement, as presently claimed, of a dispersant and no suggestion of the active ingredient to dispersant ratio. Limitations in a claim distinguishing over the prior art cannot be ignored. In re Boe, et al., 184 U.S.P.Q. 38 (C.C.P.A. 1974). Boeck et al. at column 25 and in Example 13C discloses surfactants. Burton et al. does not disclose the "inerts" included in the formulation. Boeck et al., at column 25, lines 51-63, generally discloses that an aqueous suspension could be prepared by finely grinding the compound and mixing it into a vehicle comprised of water and surfactants chosen from the types described for wettable powders. Inert ingredients such as inorganic salts and synthetic or natural gums may also be added.

Burton, et al. is consistent with Boeck, et al. in that spinosad particle size is controlled to between 2 and 6 microns (finely grinding the compound). The final composition is stated to be water based and contains no volatile organic solvents. The omission of information on the type, nature, function and amount of "inert" materials suggests particle size alone affords the physical properties of the formulation described in Burton, et al. Where the prior art gives no indication of which parameters are critical (other than particle size) and no direction as to which of many possible choices is likely to be successful, even when the claimed combination falls within the scope of possible combinations taught, does not render the claimed combination unpatentably obvious. In re O'Farrell, 7 U.S.P.Q. 2d 1673 (Fed. Cir. 1988); Ex parte Obukowicz, 27 U.S.P.Q. 2d 1063 (B.P.A.I. 1992).

Appellants contend the naphthalenesulfonate salt of Boeck et al., Example 13C should be read as within the ambit of and consistent with the disclosure; that component is a surfactant. It is impermissible within the framework of section 103 to pick and choose from any one reference only so much of it as will support a given position, to the exclusion of other parts necessary to the full appreciation of what such reference fairly suggests to one of ordinary skill in the art. In re Wesslau, 147 U.S.P.Q. 391 (C.C.P.A. 1965). Burton et al. is within the ambit of Boeck, et al. in that only a specific particle size range is disclosed and no disclosure of inerts is provided.

#### Dispersant or Surfactant

The specification in the present case provides an important defined distinction between "dispersant" and surfactant."

At page 5, line 31 through page 6, line 20, of the present specification, the term "dispersant" is discussed and defined as an agent that does not reduce the surface tension of water below 40 dynes/cm. Surfactants are defined as those agents that reduce the surface tension of water at or below 40 dynes/cm. The dispersant agents useful in the

formulations of the present invention, and surfactants that are useful in the formulations of the present invention are distinct by definition. As a person skilled in the art would appreciate, the surface tension properties of the “dispersants” and “surfactants” useful in the present invention, and the art, are separate and distinct.

As further noted on page 2 of the present specification, previous formulations of spinosyns with relatively low concentrations of dispersant, as compared to the higher concentrations in the present formulations, tended to lack homogeneity and predictability with respect to anticipated spinosyn concentrations upon dilution. The present formulations are limited to aqueous suspensions as distinguished from wettable powders, aqueous solutions and various other formulation types generally described in the Boeck et al. reference.

As described in Kirk-Othmer, Encyclopedia of Chemical Technology, 4<sup>th</sup> Ed., V. 8, p. 293, 300 (1993)(copy enclosed as Attachment 1), dispersants are primarily used to prevent settling (increase stability) of solid particles in a liquid media. Surfactants, in contrast, are used to stabilize liquid surfaces within another liquid. On solid particles, surfactants adsorb and wet the surface, but do not provide stable dispersions. Although the terms “dispersant” and “surfactant” are frequently used interchangeably, there are important differences between the two classes of materials. These differences are known to those skilled in the art. For example, surfactants tend to be small molecules containing both a hydrophilic and a hydrophobic portion and are defined by the nature of the molecule. Surfactants tend to orient at the air-water interface, oil-water interface or sometimes at a liquid-solid interface depending on the length of the hydrophobic portion and the nature of the hydrophilic part. In contrast, dispersants, which tend to be larger molecules, are defined more by their use, which is to disperse a solid in a liquid.

Appellants respectfully contend, as noted above, the terms “dispersant” and “surfactant” are on occasion used interchangeably. It does not follow that an agent termed a surfactant will be functionally useful to provide stability of solid particles in a liquid media. The Examiner’s reference to Atlox 4913 is an example of this interchangeable use. As described on page 6, line 15 of the present specification, Atlox 4913 is an example of a comb polymeric dispersant. As described in Technical Bulletin 00-4 by Uniqema (Attachment 2) Atlox 4913 is described generally as a polymeric surfactant and more particularly (page 2) as a graft or comb hydrophilic methyl methacrylate graft copolymer. Further, it is said to be “...useful as a dispersant for organic or inorganic active ingredients in water, ...” (See also Figures 5 and 6 on page 2). Unlike the Atlox random copolymer and block copolymer (4914 and 4912), both of which are useful for stabilizing liquid-liquid emulsions, particularly water in oil, Atlox 4913 is noted as “N/A” regarding emulsions. Its use (function) is as a dispersant, not a surfactant, despite the reference to it as a polymeric surfactant.

In any event, Atlox 4913 is not a condensed formaldehyde/naphthalene

sulfonic acid or salt thereof dispersant as presently claimed.

Appellants respectfully contend there is nothing in Boeck et al., or in Burton et al. to suggest the requirement of a dispersant, within the defined parameter for a dispersant, or the active ingredient to dispersant weight ratio and certainly not the particular dispersants at the particular ratio claimed by Appellants.

Data of Record

Appellants further contend the data in Example 1 of the present specification establishes the unobvious advantages of including a dispersant within the stated ratio of the presently claimed invention assuming a *prima facie* case of obviousness is found to exist. The data in Example 1 demonstrates the enhanced physical stability of the milled spinosad and dispersant (ammonium salt of sulfonated naphthalene condensate) containing formulations of the present invention and supports the statement at page 2, lines 11-12, of the specification of the surprising nature of the observations.

Appellants contend the data in the specification at Example 1 supports the nonobviousness of the presently claimed invention assuming a *prima facie* case of obviousness is found to exist. Two concentrated aqueous suspension formulation groups were prepared with one group containing a dispersant (ammonium salt of sulfonated naphthalene condensate) and one group without a dispersant. As noted at page 13, lines 17-18, the spinosad was milled to a particle size of from 3 to 7 microns. The two groups of aqueous suspensions contained 25 g/L of spinosad which is 2.5% by weight. For the dispersant containing group, the dispersant is present at about 2% by weight active dispersant on a solids basis. It should be appreciated, these amounts of dispersant are well below the naphthalenesulfonate salt amount in Boeck et al., Example 13C. The particle size is very close to the disclosure of Burton et al. These concentrated aqueous suspensions were diluted in sufficient amounts of tap or deionized water at various pH levels to afford a theoretical concentration of 100 ppm of spinosad. The samples were evaluated for actual spinosad concentration immediately upon dilution and after 24 hours.

The data in Table 1, page 14, shows, as described in the specification, that including a dispersant within the claimed range significantly improved spinosad concentrations, both initially and at 24 hours post-dilution in aqueous formulations at the stated pH levels and in both hard and soft water. The dispersant is also said to have facilitated resuspension properties of the formulation after quiescent settling of the solids from the suspension. Appellants respectfully contend this data supports the unobvious advantages of the presently claimed formulations over Boeck et al. in view of Burton et al. assuming a *prima facie* case of obviousness is found to exist.

Boeck et al. does not disclose particle size requirements or the inclusion of a dispersant and clearly not a condensed formaldehyde/naphthalene sulfonic acid or salt

dispersant at a spinosad:dispersant weight ratio of 3:1 to 1:5. Appellants contend, when taken together, despite Burton et al. disclosing a particle size of 2 to 6 microns and the Boeck et al. disclosure, the data in Example 1 supports the unobviousness and patentability of the presently claimed invention.

Summary

It is Appellants position that a combination of Boeck et al. and of Burton et al. does not establish a prima facie case of obviousness. Boeck, et al. does not suggest the requirement for a dispersant (as defined in the present specification). Burton, et al. does not disclose a dispersant. Neither of these references, taken alone or together, suggests an ectoparasiticidal aqueous suspension formulation suitable for administration to animals, including a dispersant at an active ingredient to dispersant weight ratio of 3:1 to 1:5.

Assuming, however, a prima facie case of obviousness is found to exist, the data in Example 1 clearly demonstrates the unobviousness of the formulations as presently claimed. These formulations require a condensed formaldehyde/naphthalene sulfonic acid or salt thereof dispersant (dispersant as defined) at a spinosad to dispersant weight ratio of 3:1 to 1:5.

Respectfully submitted,



John C. Demeter  
Attorney for Appellants  
Registration No. 30,167  
Phone: 317-276-3785

Eli Lilly and Company  
Patent Division/JCD  
P.O. Box 6288  
Indianapolis, Indiana 46206-6288

May 7, 2004

Appendix  
Claims on Appeal

1. (Cancelled)
2. (Previously Presented) A formulation of Claim 16 wherein the average particle size of spinosad is about 2 to about 7 microns.
3. (Previously Presented) A formulation of Claim 2 wherein the amount of spinosad is from about 0.02 to about 50 weight percent of the formulation.
4. (Previously Presented) A formulation of Claim 3 wherein the amount of spinosad is from about 2 to about 5 weight percent of the formulation.
5. (Cancelled)
6. (Cancelled)
7. (Cancelled)
8. (Previously Presented) A formulation of Claim 4 wherein the surfactant is present in an amount of from about 0.1 to about 5 weight percent of the formulation.
9. (Previously Presented) A formulation of Claim 8 wherein the spinosad is present in an amount of about 25 grams per liter of the formulation.
10. (Previously Presented) An article of manufacture, comprising packaging material and a formulation for controlling an ectoparasite infestation on a small ruminant or companion animal contained within said packaging material, wherein said formulation comprises:  
a unit dose of a formulation of Claim 16; and herein said packaging material comprises a label or package insert with instructions for administering the dose to the animal.
11. (Cancelled)
12. (Previously Presented) A method of controlling an ectoparasite infestation on a small ruminant or companion animal, comprising administering to the animal an effective amount of a formulation of Claim 16.
13. (Original) The method of Claim 12 wherein the formulation is applied to the head, neck, shoulders or back of the animal by a spot-on or pour-on protocol.
14. (Cancelled)
15. (Cancelled)
16. (Previously Presented) A stable ectoparasiticidal aqueous suspension formulation suitable for administration to animals comprising:

- a. An ectoparasitical amount of spinosad, milled to an average particle size of from 1 to 15 microns;
- b. A condensed formaldehyde/naphthalene sulfonic acid or salt thereof dispersant, wherein the spinosad:dispersant weight ratio is 3:1 to 1:5;
- c. 0.3 to 5 weight percent of a mineral thickener;
- d. an antimicrobial agent acceptable for topical veterinary applications in an amount effective to prevent microbial growth in the suspension;
- e. propylene glycol;
- f. 0.05 to 3 weight percent of xanthan gum;
- g. 0.1 to 10 weight percent of at least one surfactant;
- h. an antifoam agent; and
- i. water; wherein

said formulation is chemically and physically stable.

KIRK-OTHMER

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Deuterium  
Dewatering  
Dialysis  
Diamines  
Amines  
Diatomite  
Dicarboxylic  
Dietary Fiber  
Diffusion  
Dimensions  
Dimer Acids  
Disinfectants  
Dispersants  
Distillation  
Distillation  
Extract  
Diuretic Agents

## DISPERSANTS

Dispersants are materials that help maintain fine solid particles in a state of suspension, and inhibit their agglomeration or settling in a fluid medium. With the help of mechanical agitation, dispersants can also break up agglomerates of particles to form particle suspensions. Another use of dispersants is to inhibit the growth of crystallites in a supersaturated solution. This characteristic is also known as precipitation inhibition, threshold inhibition, or antinucleation. Overall, dispersants are useful in preventing settling, deposition, precipitation, agglomeration, flocculation, coagulation, adherence, or caking of solid particles in a fluid medium.

Specific terms used in this article to compare various states of particles that are undispersed are

**Aggregation:** a general term to describe an association of particles.

**Agglomeration:** a process where precipitation particles grow by collision with other particles. Pigment agglomerates can be broken into smaller primary particles with the aid of mechanical shear.

**Flocculation:** a relatively reversible aggregation often associated with the secondary minimum of a potential energy diagram. Particles are held together loosely with considerable surface separations.

**Coagulation:** a relatively irreversible aggregation often associated with the primary minimum of a potential energy diagram of two approaching particles. Particles are held together closely.

## Physical Chemistry of Dispersants

A convenient way to understand particle dispersion is to consider the process in four successive parts: the nature of particles and surfaces, adsorption onto particles, interface properties, and forces of attraction and repulsion.

**Particles and Surfaces.** Dispersants are primarily used to increase stability (prevent settling) of solid particles in liquid media, whereas surfactants are used more frequently to stabilize liquid (including polymer latex) surfaces within liquids. When the surface of a liquid is increased (stressed), molecules of the liquid flow to the surface to lower its energy, "healing" it. In contrast, solids exhibit no significant flow to the surface. Any stresses applied therefore remain in the form of a higher energy surface. Thus the history of a particle is important to its surface properties. Treatments that alter particle surface properties include freshly cleaving a surface along lowest energy crystal faces, adsorption of molecules and ions, heating or cooling, friction, corrosion, and grinding or polishing. The process by which a particle is formed also affects its surface properties. Examples of this include screw and spiral dislocations, missing layers, and other defects due to contamination or stress during formation (1).

**Crystals.** A crystal in equilibrium with a solution contains a point whose distance to a crystal face is proportional to the specific surface free energy of that face (Wulff theorem). If equilibrium is disturbed, the system shifts toward the condition of minimum energy by precipitating more substance on, or dissolving

dom of movement, while the solvent moves in to resolute the polymer layer. As with electrostatic repulsion, an energy barrier is created. A common approximation used is that the strength of the energy barrier rises steeply at slightly less than the adsorbed layer thickness. Some of the practical differences between sterically and electrostatically stabilized dispersions may be summarized as follows (14):

Steric stabilization	Electrostatic stabilization
insensitive to electrolyte	coagulation occurs with increased electrolyte
effective in aqueous and nonaqueous media	more effective in aqueous media
effective at high and low concentrations	more effective at low concentrations
reversible flocculation common	coagulation often irreversible
good freeze-thaw stability	freezing often induces irreversible coagulation

Advances have been made in directly measuring the forces between two surfaces using freshly cleaved mica surfaces mounted on supports (15), and silica spheres in place of the sharp tip of an atomic force microscopy probe (16). These measurements can be directly related to theoretical models of surface forces.

#### Comparisons with Other Materials

**Surfactants vs Dispersants.** Surface-active agents or surfactants (qv) are functionally related to dispersants. Surfactants are used primarily in systems where a second, nonaqueous liquid phase is present. As a result, surfactants are used to stabilize oil-water emulsions such as lubricating oils, lotions, resins, and latices. On solid particles surfactants adsorb and wet the surface, but do not yield stable dispersions due to small barrier thickness. Although the terms dispersant and surfactant are frequently confused, there are several important differences between the two classes of materials. For example, surfactants, which are small molecules containing both a hydrophilic and a hydrophobic portion, are defined by the nature of the molecule. Surfactants tend to orient at the air-water interface, oil-water interface, or sometimes at a liquid-solid interface depending on the length of the hydrophobic portion and the nature of the hydrophilic part (anionic, cationic, or nonionic). In contrast, dispersants, which tend to be larger polymeric molecules, are defined more by their use, which is to disperse a solid in a liquid. Surfactants adsorb at surfaces, preferring to be out of the water phase. Dispersants adsorb by means of chemisorption or electron transfer, using specific anchoring groups. As an example, most dispersants will not adsorb at latex surfaces, in contrast to surfactants, which do. Although there are some dispersants that have significant surfactant-like properties, a quantitative way to distinguish the two classes is by measuring their surface tension in water. A 0.02% by weight solution of a surfactant produces a surface tension of <40 mN/m (= dyn/cm), whereas in a dispersant solution it will generally be above that number.

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**Table 4. Comparison of Chelants and Flocculants**

Material
EDTA
ATMP
polycarboxylic acid <sup>a</sup>
anionic surfactant
anionic polyacrylamide <sup>b</sup>

<sup>a</sup>2000 mol wt.  
<sup>b</sup>5,000,000 mol wt.

# Atlox® Polymeric Surfactants: Agricultural Applications

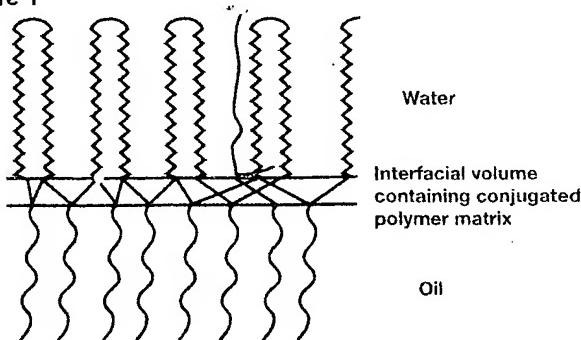
## Introduction

Atlox polymeric surfactants are medium molecular weight nonionic materials which use steric mechanisms to stabilize emulsions and dispersions. The hydrophilic portion is polyethylene oxide (PEG) and the hydrophobic portion is poly 12-hydroxystearic acid (pHSA) or alkyd resin. The mechanism of stabilization involves the solvated pendant side chains extending far into the continuous medium with the polymeric backbone adsorbing to the solid or liquid internal phase. Low levels (< 5%) of Atlox polymerics alone or in combination with nonionic or anionic wetting agents achieve excellent results. The Atlox polymerics fall into 3 categories: Random, Block copolymers and Graft copolymers. These are described below.

## Random Copolymers

Atlox 4914 is an alkyd-PEG resin with a low HLB (5-7) and good solubility in a wide range of hydrocarbons which forms water-in-oil (W/O) emulsions. When combined with Atlas® G-5000, a hydrophilic AB block copolymer, oil-in-water emulsions can be formed. Figure 1 shows the conformation of Atlox 4914 at the oil-water interface.

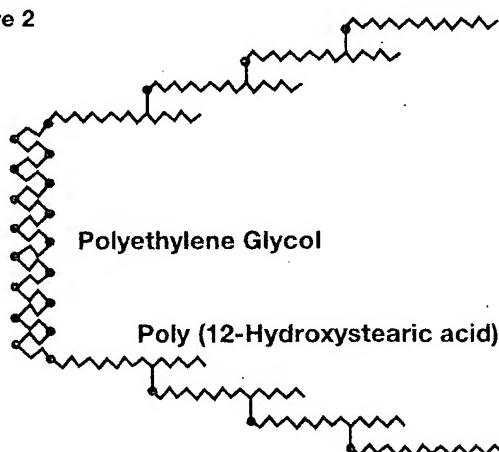
figure 1



## Block Copolymers

Atlox 4912 is an ABA block copolymer of molecular weight around 5,000 based on 12 pHSA and PEG as shown in figure 2.

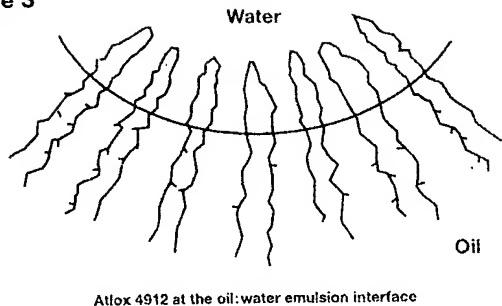
figure 2



Schematic representation of  
ordinate "ABA"block copolymer structure (Atlox 4912)

It has a low HLB (5-6) and medium polarity making it an effective W/O emulsifier (see Technical Bulletin 00-2 entitled "Atlox® 4912 Polymeric Surfactant: High Internal Phase W/O Emulsifier"). It can tolerate a high level of inorganic salts or water miscible organic materials in the aqueous phase. The oil phase can range from paraffinic mineral oils, hydrocarbon or aromatic solvents, diesel oil, kerosene, vegetable oils and fatty acid esters. The optimum use level is 5% w/w (on water) which gives a droplet size of 3 microns average diameter. These emulsions are stable to heat and high shear. Figure 3 shows the conformation of Atlox 4912 at the oil-water interface.

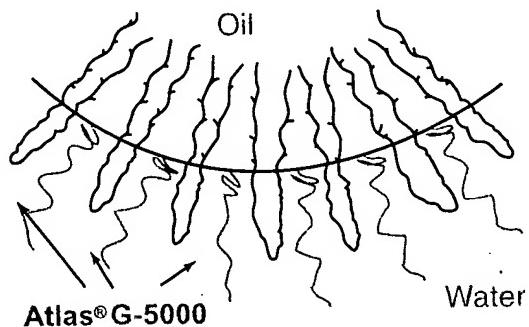
figure 3



Atlox 4912 at the oil:water emulsion interface

Atlox 4912 may also be used in combination with conventional higher HLB nonionic surfactants to give an HLB of around 10. Addition of Atlas G-5000 allows the formation of O/W emulsions

figure 4. Atlox 4912 + Atlas G-5000 packing at the interface



as illustrated in figure 4.

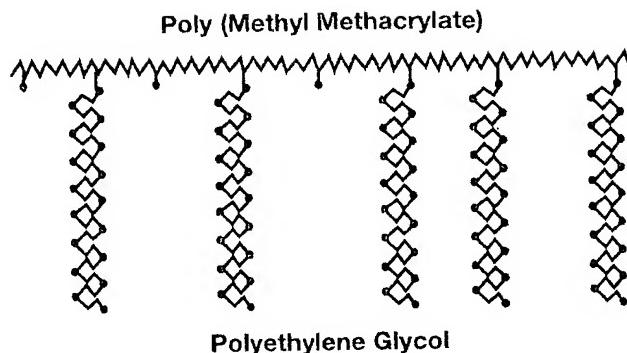
To ensure stable emulsions, a high shear mixer is used. Atlox 4912 is heated to 50°C in the oil phase. The water phase is added to the surfactant/oil blend under shear. For O/W emulsions, inversion takes place during the addition of water. Alternatively, the oil phase can be added directly to the aqueous phase under shear.

#### Graft or Comb Copolymers

Atlox 4913 is a hydrophilic methyl methacrylate graft copolymer with an HLB of around 11-12 of chemical structure shown in figure 5. It is useful as a dispersant for organic or inorganic active ingredients in water, its position at the interface is shown in figure 6.

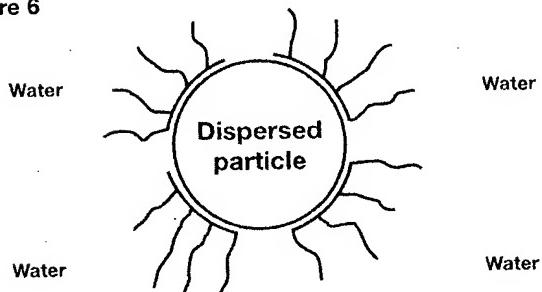
The dispersions formed using Atlox 4913 are sterically stabilized by the hydrated PEG extending from the acrylate polymer backbone and are relatively insensitive to the presence of salts or solubilized organic materials in the aqueous phase.

figure 5



Schematic representation of graft comb copolymer structure (Atlox 4913).

figure 6



#### Benefits of Atlox Polymeric:

- Improved emulsion or dispersion stability
- Aqueous or non-aqueous applications
- Low foaming
- Stable in highly ionic systems
- Nonionic surfactants for optimum compatibility with active ingredients

The use of polymeric surfactants should not simply be considered as replacing conventional anionic, cationic, amphoteric or nonionic surfactants in existing functions. For that, the price difference is not offset by the lower usage levels. The true benefit of polymeric surfactants is that they provide effects not attainable with conventional surfactants.

### **Colloidal Stabilization and Polymeric Surfactants**

The use of ionic, usually anionic, surfactants to stabilize colloidal particles by electrostatic forces is well known. People are very familiar with the application of the DLVO-theory of electrostatic stabilization. The DLVO-theory provided functional guidance to formulating work using ionic surfactants. Coupled to the fact that ionic surfactants were available at an earlier stage than ethoxylated nonionics and that they show relative cost-effectiveness in less demanding emulsions or dispersion conditions when compared with nonionic surfactants, the use of ionic surfactants is still fairly widespread.

In an environment where formulation chemists are demanding much broader applicability of surfactants and where formulations are becoming even more complex, it is steric stabilization phenomena induced by polymeric nonionic surfactants that are now becoming more important.

Steric stabilization mechanisms are not only used for stabilization of emulsions and dispersions in aqueous media. Some types of polymeric surfactants, specifically the 'Atlox' LP-and-PS series, use the same mechanisms for stabilization in non-aqueous media.

#### **The Advantages of Steric Stabilization:**

- Emulsification and dispersion effects in aqueous and non-aqueous media
- Possibility to design tailor-made molecules
- Reduced sensitivity to high electrolyte concentration.

The use of the classical ethylene oxide-propylene oxide based nonionic surfactants of the A-B or A-B-A type also has some distinct disadvantages. The most important disadvantage is the fact that desorption of these surfactants from the interface often occurs. This is due to the similarity in polarity and solubility of the alkyleneoxide groups. Additionally, solvation of ethylene oxide units is dependent on the ionic strength of the medium and the temperature. Next to this, propylene oxide units have a low solubility in hydrocarbons, especially aliphatic hydrocarbons.

Full use is made of all the advantages of ionic surfactants when polymeric surfactants are employed. Polymeric surfactants fulfill the conditions needed for effective steric stabilization, such as:

- More complete surface coverage to prevent contact between unprotected areas
- No desorption of anchoring chains during particle collision
- Maximum number of configurations possible for the stabilizing moiety during non-collision situations
- Good solvation of the stabilizing chains by the continuous medium.

The first two conditions imply a large adsorption energy for the stabilizing molecule. Even if the individual contact is weak, because of the polymeric nature of the molecule (a large number of weak contact points), the total adsorption energy is high. The presence of multiple weak anchoring points makes it more difficult for the stabilizing molecule to desorb.

### **Polymeric Surfactants in Suspension Concentrates**

With suspension concentrates the formulation chemist always has two objectives:

1. the production of highly deflocculated suspensions
2. the prevention of the formation of dilatant sediments as the suspension tends to settle under gravity.

The primary objective of producing highly deflocculated suspensions can easily be achieved with the use of 'Atlox' 4913. 'Atlox' 4913 provides a permanent steric barrier against flocculation.

As the material is completely and irreversibly covered by the dispersant the stabilized dispersion can be weakly flocculated by the addition of free (non-adsorbing) polymer such as polyethyleneoxide. This phenomenon is known as depletion flocculation. A first explanation for this type of flocculation was given by Asakura and Oosawa<sup>1</sup>. When two particles approach each other to a distance of separation smaller than the diameter of the free polymer coil, the polymer is excluded from the interparticle zone. This leads to the formation of a polymer-free zone. Owing to the lower osmotic pressure thus created in the region between the particles, an attraction force between the particles is produced. Tests have shown that at a certain concentration of free polymer,  $\phi p^+$  (critical polymer concentration), a sharp increase in yield value of the suspension concentrate can be observed. The critical polymer concentration is dependent on the molecular weight of the free polymer in as much as  $\phi p^+$  decreases with increasing molecular weight of the free polymer. The sharp increase in yield value at  $\phi p^+$  would indicate that some sort of structural change has occurred in the suspension concentrate i.e. weak flocculation of the dispersed particles. Increasing the free polymer concentration above  $\phi p^+$  leads to further increase in yield value, indicating an increase in the extent of flocculation. The increase in the extent of flocculation leads to the build-up of a structured sediment until for a certain free polymer concentration the sediment height is 100%.

Induction of depletion flocculation is only possible when no polymer can adsorb on the particle surface. In other words, when the particles are completely and irreversibly covered with surfactants like 'Atlox' 4913. The weakly flocculated structure of sediment is easy to redisperse. Depletion flocculation may be used as a way to prevent claying of suspension concentrates.

#### **Footnote:**

<sup>1</sup>Asakura, S., Oosawa, F., J. Chem. Phys., 22, 1255 (1954).

### Polymeric Surfactants in Concentrated Emulsions

The disadvantage of emulsifiable concentrates, such as skin irritation and flammability, have led formulators to produce concentrated emulsions or EW-formulations. The concentrated emulsion is a concentrated oil-in-water emulsion of a liquid or liquified low melting point lipophilic active ingredient. The formulation can contain up to 50% (W/W) active ingredient and 40% of water as external phase. To ensure good emulsion stability for this type of aqueous system over an extended period of time at both elevated and freezing temperatures, the demands on the surfactants are very severe.

Ordinary-type of polymeric surfactants like 'Atlox' 4912 are, because of their structural build-up, extremely suitable as emulsifiers for oil-in-water emulsions. 'Atlox' 4912 is an A-B-A type block copolymer made up of two poly (12-hydroxystearic acid) side chains esterified with a polyalkylene glycol centre chain. In a concentrated oil-in-water emulsion the hydrophobic PHSA-chains of 'Atlox' 4912 act as the anchoring groups into the internal oil phase and the hydrophilic, high molecular weight polyethylene glycol centre provides the stabilization in the external aqueous phase. The extension of the hydrophobic side chains into the oil phase may be several times larger than for a conventional C<sub>18</sub>-sorbitan ester.

### Polymeric Surfactants in Suspoemulsions

The step to suspoemulsions by combining a suspension concentrate with a concentrated emulsion is the next logical one. It provides the opportunity to have a formulation with multiple active ingredients while combining the advantages of both separate formulations such as:

- Low or no flammability problems owing to the use of no or low levels of solvent
- Minimal eye-and skin irritation
- Ease of incorporating adjuvants.

The challenge to formulation chemists in the optimisation of suspoemulsions is even greater. Not only is the formulator confronted with the problems of destabilization that can occur in the two separate formulation types, but by combining them he has created additional ones such as heteroflocculation and enhanced emulsion coalescence.

Heteroflocculation is the flocculation that occurs when a solid dispersed particle comes into contact with an emulsion droplet because of insufficient stabilization of the two dispersed phases. When a solid particle is wetted by an oil droplet a depletion of surfactants can occur on the oil-water interphase. If the solid particle is wetted by several oil droplets, emulsion coalescence may occur. The rate of coalescence may be increased since the solid particle acts as a sort of catalyst.

It is clear that by careful selection of surfactants it is possible to overcome the problems of heteroflocculation and enhanced emulsion coalescence. The use of an irreversibly adsorbed polymeric surfactant, such as 'Atlox' 4913, onto the solid particle to create a stable dispersion, will influence heteroflocculation in the suspo-emulsion. Also stabilizing the oil-in-water emulsion with 'Atlox' 4912 will create a suspo-emulsion that is stable against heteroflocculation and emulsion coalescence. Because of strong anchoring of the surfactant in the oil droplet and strong steric repulsion, the wetting of a suitably protected solid particle is very unlikely.

### Polymeric Surfactants in Multiple Emulsions

A multiple emulsion is a system in which one internal phase is emulsified into a second phase of different polarity, the resulting primary emulsion being further emulsified in an external phase that can be identical to or different from the initial phase. The end system is, depending on the nature of the different phases, either a water/oil/water (W/O/W) multiple emulsion or and oil/water/oil (O/W/O) multiple emulsion.

Pesticidal multiple emulsions are usually of the water-in-oil-in-water type where one or more active ingredients, soluble in the water and/or oil phase, may be incorporated. Multiple emulsions have received increasing attention as possible vehicles for the controlled release of water- or oil- soluble pesticides. Since most pesticide formulations are dilutable in water, a water-in-oil-in-water multiple emulsion can be expected to perform as a simple water-in-oil emulsion once the pesticide droplet has hit the target. The rate of release of the active ingredient will then depend on the rate at which the oil droplets burst open.

The achievement of adequate stability has, until recently, been a major limitation in the research into and the industrial application of multiple emulsions. With the use of random structure polymeric surfactants like 'Atlox' 4914, an important step has been taken to achieve the required stability of W/O/W multiple emulsions.

'Atlox' 4914 is used to stabilize the primary water-in-oil emulsion. The hydrophilic interaction is provided by multiple polar moieties such as ester-and ether-groups. The hydrophobic interactions are provided by chains of high molecular weight hydrocarbons that are well solvated by high and low polarity hydrocarbons. The secondary oil-in-water emulsion has to be stabilized by a high HLB polymeric surfactant.

### Polymeric Surfactants in Non-Aqueous Based Suspension Concentrates

In aqueous pesticide formulations the formulator can be confronted with an active ingredient that is too soluble in the aqueous phase, leading to Ostwald ripening, or perhaps with an active ingredient that is chemically unstable in water. To overcome these problems and still be able to produce a dispersed solids formulation, the formulator has to turn to non-aqueous based suspension concentrates.

The stabilization of non-aqueous suspension concentrates depends on a careful choice of surfactant. The stabilizing moiety has to be optimized so as to give excellent steric stabilization in the given external phase. Depending on the external phase a different surfactant may be needed. A special range of polymeric surfactants, the 'Atlox' LP-and PS- series, has been designed specifically for use as dispersant in non-aqueous media.

### Guide Formulations

Atlox polymeric surfactants are typically used at levels of 1-5% along with conventional anionic or nonionic surfactants. The following are examples of prototype formulas which illustrate several applications.

### Suspension Concentrates (SC)

The formulation of aqueous suspensions of pesticides requires the use of powerful dispersing agents which can keep particles in a deflocculated state by the formation of protective layers around each of them. Atlox 4913 is well adsorbed onto the solid surface because of its multiple anchoring points. Good dispersion stability is obtained because of the powerful steric repulsion between particles. Appropriate concentrations range from 1-3% of the final formulation.

#### Active A Suspension Concentrate

Active A	500 g
Atlox 4913	10-20 g
Atlox 4894	10-20 g
Propylene glycol	100 g
Thickening Agent	q.s.
Water, q.s. to	1.0 L

Procedure: This concentrate should be milled with appropriate equipment to procedure particle sizes on the order 1.5-5.0 µm.

### Concentrated Emulsions (EW)

Ordinary-type polymeric surfactants like Atlox 4912 are very useful as emulsifiers for oil-in-water emulsions. Concentrated emulsion or "EW-formulations" avoid many disadvantages of EC-formulas by reducing organic solvent levels. In the following examples, Atlox 4912 with its irreversible anchoring in the oil droplet and its strong steric repulsion between the micelles leads to the formulation of stable concentrated emulsions.

#### Active A Concentrated Emulsion

Active A solution	x g a.i./L
Active A	x g a.i.
Atlox 4914	10-20 g
Solvent to	1 L

Procedure: Add Atlox 4914 to Active A solution and mix until clear.

#### Dispersing medium

Propylene glycol	100 g
Atlas G-5000	20-30 g
Rhodopol 23 (2% mastergel)	60 g (or other thickening agent as mastergel, qs)
Water (342 ppm) to	1 L

Procedure: (A) Dissolve the surfactant and propylene glycol into water. Heat both phases to 50-60°C. (B) Slowly add Active A solution to dispersing medium and mix continuously under high shear. (C) Add the Rhodopol 23 (2% mastergel) and mix with a mechanical stirrer.

#### Suspo-Emulsions (SE)

Combining a suspension concentrate (SC) with a concentrated emulsion (EW) is even a greater challenge to the formulating chemist. The resulting suspo-emulsion has an even greater chance of destabilization by heteroflocculation or enhanced emulsion coalescence. An irreversibly adsorbed polymeric surfactant like Atlox 4913 coats the solid particles and reduces heteroflocculation by steric repulsion. Atlox 4914 can be added to the formulation to reduce emulsion coalescence.

**Active A/Active B Suspoemulsion 2:1 ratio\* g a.i./L****Active A EC: x g a.i./L**

Active A	g a.i.
Atlox 5413B	40-60 g
Atlox 4914	10-20 g
Aromatic solvent to	1 L

Procedure: Add the appropriate amount of surfactants to Active A 'blank' and mix until a clear solution is obtained.

**Active B SC: y g a.i./L**

Active B	y g a.i.
Propylene Glycol	100 g
Atlox 4894	10-20 g
Atlox 4913	10-20 g
Water (342 ppm) to	1 L

Procedure: Dissolve the surfactant in propylene glycol, add water and mix. Add Active B to the solution and mix using a mechanical stirrer. If particle size reduction is necessary for Active B, prepare the SC by wet milling (as a concentrate if necessary) with sufficient water to reach the appropriate viscosity and add the balance of water under high shear mixing when homogenous.

**Active A/Active B SE x:y ratio g a.i./L**

Active A EC	x L
Active B SC	y L
Rhodopol 23 (2% mastergel)	60 g
(or other thickening agent as mastergel, qs)	
Water (342 ppm) to	1 L

Procedure: Slowly add Active A EC to Active B SC under continuous agitation. Add the Rhodopol 23 (2% mastergel) and mix with a mechanical stirrer. If desired, the EC can be warmed to 50-55°C prior to addition into Active B SC. Additional anionic surfactant (10-20 g Atlox 4838B) can be added to the EC as necessary to adjust emulsification.

**Multiple Emulsions (W/O/W)**

Adequate stability has always been a major problem with multiple emulsions. Atlox 4914 can be used to stabilize the primary water-in-oil emulsion. A typical starting formula, shown below, consists of 80% (wt.) of primary W/O emulsion and 20% (wt.) of external water phase.

	Wt %
(A) Internal water phase	
Water	56.8
Magnesium sulphate	0.7
(B) Oil phase	
Paraffinic oil (high viscosity)	19.3
Atlox® 4914	3.2
(C) External water phase	
Water	19.0
Synperonic® PE/F127	1.0

Procedure: Heat (A) and (B) to approximately 75°C. Add (B) to (A) with high energy stirring. Cool to 30°C with moderate mechanical stirring. Add (AB) to (C) with low energy stirring until homogenous.

\*A highly loaded formulation (2:1.3 and 2.25:1.25 ratio g a.i./L) can also be produced with the same surfactant system (ratio and concentration).

### Typical Physical Properties

	Atlox 4912*	Atlox 4913**	Atlox 4914*
Polymer type	block	comb	random
Appearance	amber to brown waxy solid	pink to purple liquid	dark brown viscous liquid
Melting point °C (approx.)	40	10	25
Active content, % in solvent	95+ xylene	32+ water/PG	95+ xylene
Typical viscosity cps @ 25°C	-	6,000	65,000
Specific gravity at 25°C	0.94	1.08	1
HLB	5-6	11-12	5-7

\*Reduced or Solvent-Free versions can be made available.

\*\*Also available as water-free concentrate.

### Solubility (10% W/W) at 40°C

Solvent	Atlox 4912	Atlox 4913	Atlox 4914
Water	I	S	I
Propylene Glycol	P	S	I
Higher Glycols	I	S	I
Xylene	S	I	S
Aliphatic	S	I	S
Esters	S	S	S

S = Soluble

P = Partially soluble

I = Insoluble

### Compatibility with Various Oil/Water Systems

Surfactant	emulsion	oil phases	Aqueous phases
Atlox® 4912	W/O	Paraffinic mineral oils	Water
	OR		
	O/W*	Aromatic hydrocarbons	High ionic solutions
Atlox® 4913		Aliphatic hydrocarbons	Lower alcohol solutions
			Glycols & glycol Ethers
	N/A	Vegetable oils	Water
Atlox® 4914		Polar organic liquids	High ionic solutions
			Lower alcohol Solutions
			Glycol & glycol ethers
Atlox® 4914	W/O	Paraffinic mineral oils	Water
	OR		
	O/W*	Aromatic solvents	High ionic solutions

\*O/W Emulsions can be obtained using Atlox® 4912 and Atlox® 4914 only when used in combination with a high HLB nonionic block copolymer such as Atlox® G-5000

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